DIFFERENCES IN SOME CHEMICAL PROPERTIES OF INNERWOOD AND OUTERWOOD FROM FIVE SILVICULTURALLY DIFFERENT LOBLOLLY PINE STANDS[1]

Todd F. Shupe

Extension Specialist

Louisiana Cooperative Extension Service
Louisiana State University Agricultural Center
Baton Rouge, LA 70803

Chung Y. Hse

Principal Wood Scientist

USDA Forest Service
Southern Research Station
Pineville, LA 71360

Elvin T. Choong

Professor

Louisiana Forest Products Laboratory
School of Forestry, Wildlife, and Fisheries
Louisiana State University Agricultural Center
Baton Rouge, LA 70803

and

Leslie H. Groom

Research Technologist

USDA Forest Service
Southern Research Station
Pineville, LA 71360

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ABSTRACT

The influence of five different silvicultural management strategies on the chemical composition (extractives, Klason lignin, holocellulose, and alpha-cellulose) of loblolly pine outerwood and innerwood was investigated. Stands that were managed in a plantation setting using growth-accelerating treatments showed higher extractive contents than the other stands. Wood from the juvenile area (innerwood) yielded more extractives than outerwood (mature wood). Holocellulose and alpha-cellulose were not significantly affected by silvicultural practice but were found in a much greater concentration in outerwood than innerwood due to the greater density in the outerwood region. Klason lignin was inversely related to holocellulose.

Keywords: Alpha-cellulose, hot-water extractives, alcohol-benzene extractives, ether extractives, Klason lignin, holocellulose, silvicultural treatments, wood type.

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INTRODUCTION

Loblolly pine is the principal timber species in the South for a variety of wood-based products. Consequently, numerous investigations have been conducted to assess its chemical properties. Southern yellow pine plantations presently make up one-third of the acreage in pine forests but are projected to account for 56% of all pine stands by the year 2000. By 2030, plantations are expected to make up two-thirds of the South's pine forests (Brown and McWilliams 1990). Consequently, researchers have addressed the effect of various silvicultural practices on selected chemical properties of wood. Zobel et al. (1961) addressed the effect of fertilizer on the alpha-cellulose and holocellulose contents of loblolly pine wood. More recent research by Shupe et al. (1996) investigated the individual and interactive effects of fertilization, thinning, and pruning on the extractive content, Klason lignin, holocellulose, and alpha-cellulose contents of 12-year-old loblolly pine innerwood and outerwood.

The objectives of this study were to expand the study by Shupe et al. (1996), which was on 12-year-old loblolly pine trees, and to address the effect of various silvicultural treatments on the chemical properties of older loblolly pine trees. Specifically, the objectives of this study were: (1) to determine the effect of five different silvicultural strategies on the chemical composition of loblolly pine wood, and (2) to evaluate the differences in the chemical composition of loblolly pine innerwood and outerwood.

PROCEDURES

Plantation sampling

Five representative trees each from five silviculturally different loblolly pine (*Pinus taeda* L.) stands growing near Crossett, AR, were harvested and bucked into peeler bolts. A summary of basic stand information is presented in Table 1. All stands are described in detail by Baker and Bishop (1986). Three of the silvicultural regimes were even-aged and consisted of stand 1 (sudden sawlog), stand 2 (conventional), and stand 3 (natural regeneration). The sudden sawlog and conventional stands were the only true plantations included in the study. The uneven-aged stand investigated was subdivided into two tree age classes, i.e., stand 4 (single tree selection) and stand 5 (crop trees).

The even-aged stands can be described as follows. Stand 1 (sudden sawlog) was harvested at age 48 and was subjected to green pruning and biennial mowing. The goal of a sudden sawlog silvicultural strategy is to produce trees of sawlog dimension as rapidly as possible. Stand 2 (conventional) was 48 years old at harvest and was moderately thinned. It was never pruned or treated for understory control. This stand is typical of many pine stands throughout the South. Stand 3 (natural regeneration) was 47 to 49 years old. These trees were naturally regenerated and were never subjected to thinning, pruning, or understory control.

The mature, uneven-aged site had been under selection management for 50 years. During

TABLE 1. *Basic stand information mean values of the five harvested loblolly pine trees from the five stands growing near Crossett, AR. Five trees were harvested from each stand.*

Stand	Age (Years)	Height (ft.)	DBH[1] (in.)	Basal area (ft.²/acre)	Site index	Live crown ratio (%)[2]
1—Sudden sawlog	48	94.2	21.1	90	95	56
2—Conventional	48	93.8	15.3	118	95	39
3—Natural	48	98.6	16.4	76	100	39
4—Single tree	49	88.6	16.4	72	89	55
5—Crop tree	79	110.2	24.7	42	97	56

[1] Diameter at breast height.
[2] Live crown ratio = (length of live crown/total length of tree) × 100.

this time, two age-classes of trees developed. Stand 4 (single-tree selection) included 47- to 51-year-old dominant and codominant trees. Stand 5 (crop trees) was 77 to 85 years old and was harvested from the same stand as the single-tree selection. These two groups are hereafter referred to as separate stands for simplicity even though both were actually growing together. The crop trees (stand 5) had been left uncut by all previous thinning operations and were easily separated from the single tree selection group (stand 4) based on size.

This study was done in conjunction with other veneer-based studies. Consequently, the bolts were rotary-peeled by Hunt Plywood at Pollock, LA, to approximately 137 cm × 249 cm at a target thickness of 0.3175 cm. The veneer was coded according to stand, tree number, and bolt number as it was peeled. The veneer was dried commercially to a moisture content (MC) of 6–8%, transported to the USDA-Forest Service, Southern Research Station in Pineville, LA, stored in a controlled environment of 72°F and 36% relative humidity (RH), and graded by an APA-The Engineered Wood Association veneer grader.

Veneer sampling was limited to the bottom two peeler bolts for all stands. Innerwood was considered the last ten veneer sheets removed from a peeler bolt, and outerwood was treated as the first ten sheets peeled from a bolt. All bolts were peeled to a final diameter of 7.62 cm. Therefore, our innerwood was considered to be entirely juvenile wood and heartwood, and the outerwood was clearly in the sapwood zone. Clear wood that passed through a laboratory Wiley Mill until it would pass through a No. 40 mesh sieve yet also be retained on a No. 60 mesh sieve was used for chemical property testing.

TABLE 2. *Summarized means of loblolly pine outerwood and innerwood alcohol-benzene, ether, and hot-water extractive contents from five different silvicultural treatments.*

	Extractive content (%)			
Stand[1]—wood type	Alcohol-benzene	Ether	Hot-water	Total
1—Outerwood	4.53[2] A[3]	0.50 A	3.97 A	9.00 A
2—Outerwood	4.03 A	0.42 A	3.93 A	8.38 A
3—Outerwood	3.39 B	0.39 A	3.35 B	7.13 B
4—Outerwood	2.58 C	0.41 A	3.22 B	6.21 B
5—Outerwood	2.50 C	0.40 A	3.15 B	6.05 B
1—Innerwood	6.98 A	0.53 A	5.01 A	12.52 A
2—Innerwood	6.92 A	0.55 A	4.75 A	12.22 A
3—Innerwood	5.49 B	0.42 A	3.82 A	9.73 B
4—Innerwood	5.32 B	0.41 A	3.53 B	9.26 B
5—Innerwood	5.23 B	0.39 A	3.23 B	8.85 C

[1] Stand 1 = Sudden sawlog. Stand 2 = Conventional. Stand 3 = Natural regeneration. Stand 4 = Single tree selection. Stand 5 = Crop trees.
[2] Represents the mean of 12 samples.
[3] Within either wood type grouping, means followed by the same letters indicate no significant difference exists between means for a particular property according to the Tukey mean separation procedure. Significant differences were declared at $\alpha = 0.05$. The grouping "A" pertains to the group with the highest mean value, B to the group with the second highest mean values, and C to the third highest mean values.

Laboratory experimentation and data analysis

Chemical constituent values were obtained using the following test procedures: (1) alcohol-benzene extractive content (ASTM D 1105-84), (2) hot-water extractive content (ASTM 1110-84), (3) ether extractive content (ASTM 1108-84), (4) Klason lignin content (D 1106-84), (5) holocellulose content (ASTM D 1104-56), and (6) alpha-cellulose content (ASTM D 1103-60) (ASTM 1982, 1993).

The statistical analysis was conducted using SAS programming methods (SAS 1989) in conjunction with analysis of variance (AOV) techniques (Steel and Torrie 1980; Box et al. 1978). The significance of each factor and factor interactions were determined at the $\alpha = 0.05$ level using Type III Sum of Squares. It was determined that samples were normally distributed with different means and with a common variance.

RESULTS AND DISCUSSION

Extractives (extraneous material)

Extractive content values obtained by three methods using innerwood and outerwood from the five silviculturally different stands are presented in Table 2. Tukey's mean separation letters are listed next to each mean value.

In the outerwood region, stand 1 (sudden sawlog) and stand 2 (conventional) gave significantly higher alcohol-benzene and hot-water extractive contents than the slower grown trees from stand 3 (natural), stand 4 (single tree), and stand 5 (crop tree). Mean ether extractive content values ranged from 0.39%–0.50% and showed no significant differences (Table 2). These findings are comparable with those of Shupe et al. (1996), who found mean outerwood extractive values of 2.90%, 0.41%, and 3.18% for alcohol-benzene, ether, and hot water, respectively.

A similar pattern was detected for innerwood extractive contents. Again, stand 1 (sudden sawlog) and stand 2 (conventional) gave significantly greater alcohol-benzene and hot-water extractives. Stand 3 (natural) gave a statistically similar mean value to stand 1 (sudden sawlog) and stand 2 (conventional) for hot-water extractives. No differences were detected for ether extractives due to the small range (0.39%–0.55%).

Our values are greater than those reported by Max (1945) for alcohol-benzene (2.76%) and hot water (1.24%) on green loblolly pine wood. However, Max (1945) found a much greater value for ether extractives of 1.83%. It is interesting to note that all of our values for innerwood are much greater than those reported by Max (1945) even though our wood was dried prior to extraction, which typically reduces the amount of extractives removed by the alcohol-benzene or ether extraction methods. Pettersen (1984) reported the following mean extractive contents for loblolly pine: 1% NaOH (11%), hot water (2%), and alcohol-benzene (3%).

Our findings regarding higher extractive contents for stand 1 (sudden sawlog) and stand 2 (conventional) are logical because Kramer and Kozlowski (1979) have shown that extractives are products of metabolic tree growth. Therefore, silvicultural treatments that serve to increase tree growth and vigor should increase the extractive content. This hypothesis was not directly tested in our study, but it appears to be valid in this study because stand 1 (sudden sawlog) and stand 2 (conventional) were the only plantations sampled and were actively managed for rapid growth. In general, Shupe et al. (1996) found this hypothesis not to be true because they found minimal and inconsistent differences due to fertilization, pruning, and stand density using the same three methods of determination of extractive content.

As expected, the hot water and alcohol-benzene extractive contents were significantly greater in innerwood than outerwood. Naturally, the innerwood wood type contained a higher percentage of extractives than the outerwood wood type. The difference in southern pine heartwood (high extractive concentration) and sapwood (low extractive concentration) extractive content has been well documented (Ritter and Fleck 1926; Wahlenberg 1960; Posey and Robinson 1969; McMillin 1968; Hillis 1987). The mean values for ether extractives showed little variation between innerwood and outerwood in this study and also in the Shupe et al. (1996) study.

Nonextraneous material

The mean Klason lignin, holocellulose, and alpha-cellulose contents obtained from the innerwood and outerwood portions of five silviculturally different stands are presented in Table 3. In contrast to the extractive content findings, the slower-grown stands gave higher mean values for both innerwood and outerwood. Specifically, stand 4 (single tree) and stand 5 (crop tree) yielded significantly higher mean values for holocellulose and alpha-cellulose for both innerwood and outerwood wood types. Holocellulose values ranged from 70.37%–74.53% for outerwood and showed no significant differences between the stands. In the innerwood region, stand 4 (single tree) and stand 5 (crop tree) yielded significantly less holocellulose than the other stands. The lower holocellulose content was expected since these stands (4 and 5) gave comparatively higher values for Klason lignin. The procedures used allow for a summative analysis of total polysaccharide and non-polysac-

TABLE 3. *Summarized means of loblolly pine outerwood and innerwood Klason lignin, holocellolose, alpha-cellulose from five different silvicultural treatments. Values are percentages of extractive-free oven-dried wood.*

	Non-extraneous material (%)		
	Holo-cellulose	Klason lignin	Alpha-cellulose
1—Outerwood	74.53[2] A[3]		46.98 B
2—Outerwood	73.39 A		43.68 B
3—Outerwood	73.33 A		42.37 B
4—Outerwood	71.32 A		49.68 A
5—Outerwood	70.37 A		50.98 A
1—Innerwood	79.79 A		40.32 B
2—Innerwood	78.63 A		38.38 C
3—Innerwood	78.50 A		37.32 C
4—Innerwood	75.61 B		44.33 A
5—Innerwood	74.43 B		45.32 A

[1] Stand 1 = Sudden sawlog. Stand 2 = Conventional. Stand 3 = Natural regeneration. Stand 4 = Single tree selection. Stand 5 = Crop trees.

[2] Represents the mean of 12 samples.

[3] Within either wood type grouping, means followed by the same letters indicate no significant difference exists between means for a particular property according to the Tukey mean separation procedure. Significant differences were declared at $\alpha = 0.05$. The grouping "A" pertains to the group with the highest mean value, B to the group with the second highest mean values, and C to the third highest mean values.

charide structural material. On an oven-dried wood basis, the holocellulose and Klason lignin contents should sum to 100% but can vary due to the alcohol-benzene extractive content and the destructive nature of the testing (Shupe 1993).

Most growth-accelerating silvicultural treatments reported in the literature have failed to significantly influence Klason lignin, holocellulose, or alpha-cellulose. However, Shupe et al. (1996) did find a significantly higher Klason lignin content with wood from a fertilized plot than from an unfertilized plot. Zobel et al. (1961) failed to detect a significant difference between heavily and moderately fertilized 25-year-old loblolly pine plantations and a control plantation for water-resistant carbohydrates, "holocellulose," and alpha-cellulose. Moreover, Shupe et al. (1996) found fertilization and pruning to have an insignificant effect and stand density to have a minimal effect on holocellulose and alpha-cellulose. Therefore, we can conclude that most forms of silvicultural manipulation have a minimal effect on the nonextraneous content of loblolly pine wood. The concentration of holocellulose and alpha-cellulose tends to closely parallel the natural increase in wood density from pith to bark as described by Megraw (1985). As wood specific gravity increases during the juvenile period, so will the holocellulose and alpha-cellulose concentration because these compounds are the primary constituents of the cell wall.

We found lower values in innerwood for Klason lignin and alpha-cellulose. Many previous investigations have found that loblolly pine outerwood possesses more holocellulose and alpha-cellulose than innerwood (Byrd et al. 1965; McMillin 1968; Stamm and Sanders 1966; Zobel and McElwee 1958; Zobel et al. 1966). These differences between innerwood and outerwood can be attributed largely to the relationship between wood density and the structural cell-wall material (i.e., holocellulose and alpha-cellulose) (Panshin and de Zeeuw 1980). Koch (1972) associated the changes in polysaccharide content across a tree diameter to the presence of juvenile wood. In short, juvenile wood, located primarily in the innerwood region, is less dense than mature wood, located largely in the outerwood region. The density of wood is determined by the amount of structural cell-wall material present. Therefore, since juvenile wood is less dense than mature wood, it follows that it also contains less holocellulose and alpha-cellulose than mature wood.

CONCLUSIONS

This research was initiated to determine the effect of different silvicultural treatments on the chemical properties of loblolly pine innerwood and outerwood. Based upon this research, the following conclusions are offered.

(1) Stands that have been managed using growth-accelerating treatments in a plantation setting showed higher extractive contents than the other stands.

(2) Wood produced during the juvenile period (innerwood) when a tree is exhibiting its most vigorous growth displayed a greater extractive content than mature wood (outer-

wood), which is produced after tree growth and vigor have declined.

(3) Holocellulose and alpha-cellulose are minimally affected by silvicultural strategies. The concentration of these chemicals mimics the natural pattern of wood density from pith to bark. Therefore, any cultural treatment that affects this natural pattern will be manifested in the polysaccharide content.

(4) Klason lignin showed an inverse relationship with holocellulose. This is logical since the total polysaccharide and non-polysaccharide structural material should sum to 100%. Therefore, an increase in holocellulose will cause a decrease in Klason lignin.

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